# REPORT DOCUMENTATION PAGE

Form Approved OMB NO. 0704-0188



| 1. REPORT DATE (DD-MM-YYYY) | 2. REPORT TYPE | 3. DATES COVERED (From - To) |
|---|---|---|
| 19-02-2015 | Final Report | 15-Feb-2014 - 14-Feb-2015 |

4. TITLE AND SUBTITLE

Final Report: Acquisition of a SAXS Facility for the Study of Novel Polymer Nanocomposite Membranes

5a. CONTRACT NUMBER
W911NF-14-1-0076

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
206022

6. AUTHORS

David Suleiman

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAMES AND ADDRESSES

University of Puerto Rico at Mayaguez
R & D Center
Call Box 9000
Mayaguez, PR 00681 -9000

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS (ES)

U.S. Army Research Office
P.O. Box 12211
Research Triangle Park, NC 27709-2211

10. SPONSOR/MONITOR'S ACRONYM(S)
ARO

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
64741-CH-REP.6

12. DISTRIBUTION AVAILIBILITY STATEMENT

Approved for Public Release; Distribution Unlimited

13. SUPPLEMENTARY NOTES

The views, opinions and/or findings contained in this report are those of the author(s) and should not contrued as an official Department of the Army position, policy or decision, unless so designated by other documentation.

14. ABSTRACT

A state-of-the-art small angle x-ray scattering (SAXS) instrument with wide angle x-ray scattering (WAXS) and grazing incidence x-ray scattering (GI-SAXS) capability was purchased and installed in the R& D Center (Lab # GB-03 of) of the University of Puerto Rico at Mayaguez (Figure 1). The Anton-Paar SAXSpace equipment was properly installed and the training was conducted during August 18-22, 2014. The equipment was extensively used contributing to several publications described ahead. These non-invasive scattering techniques provide valuable information at the nanoscale regarding: size, shape, morphology and even particle interaction. This facility will

15. SUBJECT TERMS

Nanostructure, Scattering, Polymer Nanocomposite Membranes

| 16. SECURITY CLASSIFICATION OF: | | | 17. LIMITATION OF ABSTRACT | 15. NUMBER OF PAGES | 19a. NAME OF RESPONSIBLE PERSON |
|---|---|---|---|---|---|
| a. REPORT | b. ABSTRACT | c. THIS PAGE | | | David Suleiman |
| UU | UU | UU | UU | | 19b. TELEPHONE NUMBER 787-309-7126 |

## Report Title

Final Report: Acquisition of a SAXS Facility for the Study of Novel Polymer Nanocomposite Membranes


## ABSTRACT

A state-of-the-art small angle x-ray scattering (SAXS) instrument with wide angle x-ray scattering (WAXS) and grazing incidence x-ray scattering (GI-SAXS) capability was purchased and installed in the R& D Center (Lab # GB-03 of) of the University of Puerto Rico at Mayaguez (Figure 1). The Anton-Paar SAXSpace equipment was properly installed and the training was conducted during August 18-22, 2014. The equipment was extensively used contributing to several publications described ahead. These non-invasive scattering techniques provide valuable information at the nanoscale regarding: size, shape, morphology and even particle interaction. This facility will further advance the understanding of the resulting nanostructure of novel polymer nanocomposite membranes (PNMs) at the University of Puerto Rico at Mayaguez (UPRM). It supports our current efforts with novel polymer chemistries and architectures, which incorporate functionalized organic/inorganic additives creating unique PNMs. In addition, it can also be used in many other current research efforts at UPRM such as: pharmaceutical engineering, biological engineering, catalysis, adsorption, and the development of other complex nanomaterials. This facility is the only facility in UPRM and in Puerto Rico capable of performing these powerful characterization techniques. In addition, it supports the education of numerous underrepresented Hispanic graduate and undergraduate students in STEM at UPRM, where knowledge and application of scattering techniques was very limited (only through interactions with collaborators outside Puerto Rico).


---

## Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

### (a) Papers published in peer-reviewed journals (N/A for none)

| Received | Paper |
|---|---|

**TOTAL:**

**Number of Papers published in peer-reviewed journals:**

---

### (b) Papers published in non-peer-reviewed journals (N/A for none)

| Received | Paper |
|---|---|

**TOTAL:**

**Number of Papers published in non peer-reviewed journals:**

## (c) Presentations

1. A. Ortiz and D. Suleiman. "Polymer-Titania Nanoparticles for Energy Efficient." Poster Presentation at the 2015 MRS Spring National Meeting, San Francisco, CA, April, 2015.

2. A. Millet and D. Suleiman "Sulfonation and Characterization of Poly(1,4-phenylene ether-ether-sulfone) (PEES) and Polyphenylsulfone (PPSF) for Fuel Cells and Specialty Separation Applications." Poster Presentation at the 2015 ACS PRISM Meeting San Juan, PR March, 2015.

3. D. Suleiman "Polymer Nanocomposites: Technology for the XXI Century." Oral Key Note Presentation at the 2014 Science & Materials Assembly. Mayaguez, PR, November, 2014.

4. M. Pérez and D. Suleiman Transport Properties of Sulfonated Poly(ether ether ketone) Membranes with Counter-Ion Substitution". Poster Presentation at the 2014 AIChE National Meeting, Atlanta, GA, November, 2014.

5. M. Pérez and D. Suleiman. "Synthesis and Characterization of Poly(styrene-isobutylene-methyl vinyl ether) for Direct Methanol Fuel Cell Applications". Poster Presentation at the 2014 AIChE National Meeting, Atlanta, GA, November, 2014.

**Number of Presentations:** 5.00

## Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

| Received | Paper |
|---|---|

**TOTAL:**

**Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):**

## Peer-Reviewed Conference Proceeding publications (other than abstracts):

| Received | Paper |
|---|---|

**TOTAL:**

**Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):**

## (d) Manuscripts

| Received | | Paper |
|---|---|---|
| 02/19/2015 | 5.00 | Maritza P¬erez-P¬erez, Edward M. A. Guerrero-Guti¬errez, David Suleiman. Synthesis and characterization of sulfonated fluorinated blockcopolymer membranes with different esterified initiators for DMFCapplications, Journal of Applied Polymer Science (11 2004) |
| 12/31/2014 | 1.00 | Maritza Pérez-Pérez , David Suleiman, Edward M.A. Guerrero-Gutiérrez. Synthesis and Characterization of Sulfonated Fluorinated Block Copolymer Membranes with Different Esterified Initiatorsfor DMFC Applications, Journal of Applied Polymer Science (11 2014) |
| 12/31/2014 | 2.00 | Edward M. A. Guerrero-Gutiérrez, Maritza Pérez-Pérez , David Suleiman. Morphology and Transport Properties of Sulfonated Fluoroblock Copolymer Blend Membranes, Journal of Membrane Science (12 2014) |
| 12/31/2014 | 3.00 | Maritza Pérez-Pérez , David Suleiman. Transport Properties of Sulfonated Poly(ether ether ketone) Membranes with Counter-Ion Substitution, Journal of Membrane Science (12 2014) |
| **TOTAL:** | | **4** |

**Number of Manuscripts:**

## Books

| Received | | Book |
|---|---|---|
| **TOTAL:** | | |

| Received | Book Chapter |
|---|---|

**TOTAL:**

## Patents Submitted

## Patents Awarded

## Awards

Distinguished Professor of Chemical Engineering, University of Puerto Rico (May, 2014)

## Graduate Students

| NAME | PERCENT_SUPPORTED |
|---|---|
| **FTE Equivalent:** | |
| **Total Number:** | |

## Names of Post Doctorates

| NAME | PERCENT_SUPPORTED |
|---|---|
| **FTE Equivalent:** | |
| **Total Number:** | |

## Names of Faculty Supported

| NAME | PERCENT_SUPPORTED | National Academy Member |
|---|---|---|
| David Suleiman | 0.11 | |
| **FTE Equivalent:** | **0.11** | |
| **Total Number:** | **1** | |

## Names of Under Graduate students supported

| NAME | PERCENT_SUPPORTED |
|---|---|
| **FTE Equivalent:** | |
| **Total Number:** | |

## Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

| | |
|---|---|
| The number of undergraduates funded by this agreement who graduated during this period: | 4.00 |
| The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields: | 8.00 |
| The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields: | 4.00 |
| Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale): | 7.00 |
| Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering: | 0.00 |
| The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense | 0.00 |
| The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: | 0.00 |

## Names of Personnel receiving masters degrees

NAME

**Total Number:**

## Names of personnel receiving PHDs

NAME
Edward M.A. Guerrero Gutierrez
**Total Number:** **1**

## Names of other research staff

NAME PERCENT_SUPPORTED

**FTE Equivalent:**
**Total Number:**

## Sub Contractors (DD882)

## Inventions (DD882)

## Scientific Progress

See Attachment

## Technology Transfer

**Acquisition of a SAXS Facility for the Study of Novel Polymer Nanocomposite Membranes**
**Interim Progress Report (IPR), Year 1: Scientific Progress and Accomplishments,**
**15 Feb 2014 – 14 Feb 2015, David Suleiman, PI (University of Puerto Rico at Mayaguez)**
**W911NF-14-10076**

**Project Summary:**

A state-of-the-art small angle x-ray scattering (SAXS) instrument with wide angle x-ray scattering (WAXS) and grazing incidence x-ray scattering (GI-SAXS) capability was purchased and installed in the R& D Center (Lab # GB-03 of) of the University of Puerto Rico at Mayaguez (Figure 1). The Anton-Paar SAXSpace equipment was properly installed and the training was conducted during August 18-22, 2014. The equipment was extensively used contributing to several publications described ahead. These non-invasive scattering techniques provide valuable information at the nanoscale regarding: size, shape, morphology and even particle interaction. This facility will further advance the understanding of the resulting nanostructure of novel polymer nanocomposite membranes (PNMs) at the University of Puerto Rico at Mayaguez (UPRM). It supports our current efforts with novel polymer chemistries and architectures, which incorporate functionalized organic/inorganic additives creating unique PNMs. In addition, it can also be used in many other current research efforts at UPRM such as: pharmaceutical engineering, biological engineering, catalysis, adsorption, and the development of other complex nanomaterials. **This facility is the only facility in UPRM and in Puerto Rico capable of performing these powerful characterization techniques.** In addition, it supports the education of numerous underrepresented Hispanic graduate and undergraduate students in STEM at UPRM, where knowledge and application of scattering techniques was very limited (only through interactions with collaborators outside Puerto Rico).


**Figure 1: SAXS Facility in the R&D Center (Lab # GB-03) of the University of Puerto Rico at Mayaguez**

**Students Impacted:**
Underrepresented Hispanic PhD Students in Chemical Engineering that are currently trained to use the equipment:

1. Edward M.Guerrero, **PhD obtained December, 2014**
2. Ariangelis Ortiz
3. Maritza Pérez
4. Karren Barrios

Underrepresented Hispanic undergraduate (BS) students in Chemical Engineering that have been using the equipment:

1. Rinaldo Díaz
2. Arnaldo López
3. Alexander Millet
4. Nataira Pagán
5. Luis Sotomayor
6. Vanessa Torres
7. Lorena Cruz
8. Edwin Torres

**Technical Impact**
Several research publications have been submitted by the time of completing this report. This award was acknowledged in the publications:

1. E.M.A. Guerrero-Gutiérrez, M. Pérez-Pérez, and D. Suleiman. *"Synthesis and Characterization of Sulfonated Fluorinated Block Copolymer Membranes with Different Esterified Initiators for DMFC Applications". Journal of Applied Polymer Science, 132, 42046,* **2015**.

2. M. Pérez-Pérez, and D. Suleiman. *"Transport Properties of Sulfonated Poly(ether ether ketone) with Counter-Ion Substitution". Submitted to the Journal of Membrane Science,* **2014**.

3. E.M.A. Guerrero-Gutiérrez, M. Pérez-Pérez, and D. Suleiman. *"Morphology and Transport Properties of Sulfonated Fluoroblock Copolymer Blend Membranes". Submitted to the Journal of Membrane Science,* **2014**.

4. A. Ortiz-Negrón, and D. Suleiman. *"The Effect of $TiO_2$ Nanoparticles on the Properties of Sulfonated Block Copolymer Membranes". Submitted to the Journal of Applied Polymer Science,* **2015**.

In addition to the previously submitted publications, several additional publications are expected to be completed this year as a consequence of the additional understanding provided by this equipment (this award). The next publications are expected to be higher impact factor publications (e.g., ACS Nano-12.033, Nano Letters-12.94) due to the more in-depth understanding of the resulting nanostructure provided by the 3D GI-SAXS capability. The GI-SAXS capability is currently being fully utilized with help from the University of Washington (L. Pozzo).